(100 mg, 0.2 mmol) in pyridine (2 mL) was added *p*-chlorobenzoyl chloride (0.03 mL, 0.23 mmol). The mixture was then left at room temperature for 6 h, treated with water (0.3 mL) for 5 min, and evaporated. The residue was partitioned between ethyl acetate (20 mL) and water (5 mL). TLC showed the presence of a small amount of another faster moving substance (most probably N^3-*p*-chlorobenzoyl derivative of **6a**). The ethyl acetate extract was heated in 95% pyridine at 110 °C for 2 h and cooled. The mixture was evaporated and repeatedly coevaporated with ethanol, and the residue was recrystallized from a mixture of ethanol and acetone to give 93 mg of needles of mp 245–247 °C, identical with the above-obtained sample of **6a** in all respects.

1-(5′-*O*-*p*-Chlorobenzoyl-3′-*O*-*p*-methylbenzoyl-β-D-arabinofuranosyl)4-thiouracil (4b). A mixture of **4a** (800 mg, 1.595 mmol) and phosphorus pentasulfide (710 mg, 3.19 mmol) in pyridine (25 mL) was stirred at 105 °C for 2 h and 20 min. Further phosphorus pentasulfide (300 mg) was added and the reaction continued for an additional 2 h. After cooling, the reaction mixture was partitioned between ethyl acetate (100 mL) and water (30 mL). The separated ethyl acetate layer was evaporated, the residual gum heated in water (50 mL) at 90–95 °C for 10–15 min, and the water decanted off. This procedure was repeated four times. The finally obtained solid residue was crushed with hot water, collected by suction, and recrystallized from acetonitrile to give 550 mg (67%) of **4b,** mp 254–256 °C; UV λ_{max}^{MeOH} 238 (ε 48 600) and 328 nm (ε 25 100).

Anal. Calcd for $C_{24}H_{21}N_2O_7SCl$: C, 55.77; H, 4.10; N, 5.42. Found: C, 55.64; H, 4.08; N, 5.67.

1-(2′,5′-di-*O*-*p*-Chlorobenzoyl-3′-*O*-*p*-methylbenzoyl-β-D-arabinofuranosyl)-4-thiouracil (6b). A mixture of **4b** (200 mg, 0.388 mmol) and sodium benzoate (224 mg, 1.55 mmol) in DMF (4.8 mL) was stirred at 115–120 °C for 3.5 h. After evaporation of the solvent, the residue was partitioned between ethyl acetate (50 mL) and water (10 mL). The separated organic phase was dried and evaporated, and the residue was triturated with chloroform to give 44 mg of the starting material. TLC with the filtrate using chloroform/ethyl acetate (3:1) showed the presence of a main (starting material) and two minor spots, one of which was faster moving and the other slower moving than the starting material. The filtrate was concentrated, charged on a silica gel plate (5 × 20 cm), and developed with chloroform. After usual workup, 20 mg (7.7%) of **6b,** mp 179–181 °C (from acetone + MeOH), was obtained: UV λ_{max}^{MeOH} 238 (ε 59 400) and 327 nm (ε 19 100).

Anal. Calcd for $C_{31}H_{24}N_2O_9SCl$: C, 55.45; H, 3.60; N, 4.17. Found: C, 55.21; H, 3.85; N, 4.15.

Additional starting material (56 mg) was recovered. The slower moving product was neglected.

1-(5′-*O*-*p*-Chlorobenzoyl-3′-*O*-*p*-methylbenzoyl-β-D-arabinofuranosyl)3-methyluracil (9). A mixture of **4a** (300 mg, 0.615 mmol) and *N,N*-dimethylformamide dimethylacetal (0.3 mL, 3 mmol) in chloroform (10 mL) was heated to a reflux for 4 h and cooled. The mixture was evaporated, charged on a silica gel plate (20 × 20 cm), and developed twice with chloroform/ethyl acetate (3:1). Elution of the main band with acetone gave 173 mg of a homogeneous solid, which was recrystallized from methanol to give 163 mg (52.7%) of **9** as needles of mp 173–175 °C: UV λ_{max}^{MeOH} 240 (ε 46 100) and 262 nm (ε 17 100).

Anal. Calcd for $C_{25}H_{23}N_2O_8Cl$: C, 58.31; H, 4.50; N, 5.44. Found: C, 58.54; H, 4.77; N, 5.43.

1-(5′-*O*-*p*-Chlorobenzoyl-2′,3′-di-*O*-*p*-methylbenzoyl-β-D-arabinofuranosyl)-3-methyluracil (10). Method A. A mixture of **9** (163 mg, 0.318 mmol) and sodium *p*-methylbenzoate (202 mg, 1.27 mmol, 4 molar excess) in DMF (4 mL) was stirred at 125–130 °C for 3.5 hr. TLC with an aliquot of the reaction mixture revealed the starting material as the major component with two minor products, one of which was faster moving and the other slower moving. Thus, the general pattern was similar with the case of the reactions between **4a,b** and the basic catalysts. The mixture was evaporated and the residue partitioned between ethyl acetate (30 mL) and water (7 mL). The obtained ethyl acetate extract was charged on a silica gel plate (20 × 20 cm) and developed with chloroform/ethyl acetate (3:1). The most mobile band gave 26 mg (12.9%) of **10** as needles of mp 182–184 °C after crystallization from a mixture of methanol and acetone: UV λ_{max}^{MeOH} 242 (ε 65 400) and 262 nm (ε 17 600).

Anal. Calcd for $C_{33}H_{29}N_2O_9Cl$: C, 62.61; H, 4.62; N, 4.43. Found: C, 62.43; H, 4.58; N, 4.53.

The major fraction gave 85 mg (51%) of the starting material. The other minor product was neglected.

Method B. A mixture of **9** (0.142 g, 0.277 mmol) and sodium benzoate (160 mg, 1.11 mmol, 4 molar excess) in DMF (4.5 mL) was stirred at 125–130 °C for 3.5 h. The reaction was worked up as described in method A to give 21 mg (12%) of **10,** identical with the product obtained above in terms of infrared and ultraviolet spectroscopy and mixef fusion. The other components were neglected.

Acknowledgments. The authors are grateful to Kyowa Fermentation Co., Ltd., for a generous gift of uridine.

Registry No.—**1,** 59211-02-8; **2,** 64114-40-5; **3,** 64114-41-6; sodium *p*-methylbenzoate, 17264-54-9; potassium *p*-methylbenzoate, 16518-25-5; *p*-chlorobenzoylchloride, 122-01-0; phosphorus pentasulfide, 1314-80-3; sodium benzoate, 532-32-1; *N,N*-dimethylformamidedimethylacetal, 4637-24-5.

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Cyclocarbonylation of 2-*exo*-Ethynyl-7-*syn*-norbornanol to an α-Methylene δ-Lactone

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Although α-methylene butyrolactones are much more prevalent in natural products and have thus received more synthetic attention,[2] naturally occurring α-methylene valerolactones are also known, e.g., in vernolepin and vernomenin.[3] We have thus investigated the usefulness of our $PdCl_2$/thiourea catalyst system[4] in the synthesis of α-methylene valerolactones from carbon monoxide and appropriately substituted 4-pentynols.[5]

Only traces of α-methylene δ-valerolactone (1) were obtained by this method from 4-pentyn-1-ol itself, either under catalytic conditions or in the presence of 1 equiv of $PdCl_2$; most of the starting ethynyl alcohol remained unreacted even after 60 h. However, better results seemed likely with a fused-ring system where the ethynyl and hydroxyl groups were fixed in the appropriate geometry for lactone ring formation. A suitable substrate, **2a,** proved available from the treatment

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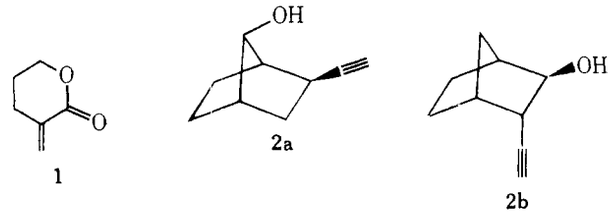
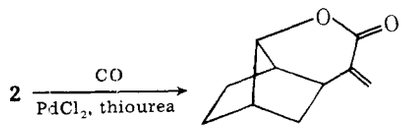


1 2a 2b

of *exo*-norbornene oxide (3) with dimethylethynylaluminum etherate.[6]

Results and Discussion

On the basis of decoupling experiments revealing an NMR coupling constant of about 3 Hz between the hydroxyl-substituted methine proton and the ethynyl-substituted methine proton, the product 2 of ethynylaluminum treatment of *exo*-norbornene oxide had originally been assigned the structure **2b.** However, **2b** would not be expected to form a lactone

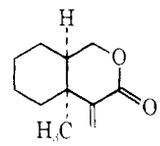


4

readily, whereas carbonylation of **2** in the presence of $PdCl_2$/thiourea gives a methylene lactone (**4**) in good yield.

The spectroscopic properties of **4** clearly show that it is an α-methylene δ-lactone and not an α-methylene γ-lactone. For example, the ν_{CO} of **4** (1722 cm^{-1}) is well below the range (1770–1750 cm^{-1}) typical of the latter,[2,7] while within the range (1730–1710 cm^{-1}) typical of the former.[5]

The ^1H NMR spectrum of **4** confirms the δ-lactone structure. The *exo*-methylene group appears as a pair of doublets (each with J = 1.3 Hz) at δ 5.94 and 5.28. Spin-decoupling shows that this J is a *geminal* coupling constant; no coupling to other, e.g., allylic, protons is resolvable. While geminal couplings are typically negligible in α-methylene γ-lactones[2,7,8] (e.g., <0.2 Hz in a case analyzed in detail in reference 7a), they are frequently observed with a value of about 1 Hz in α-methylene γ-lactones (e.g., in **5**[5c] and **1**[5d]). On the other



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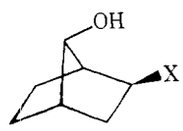
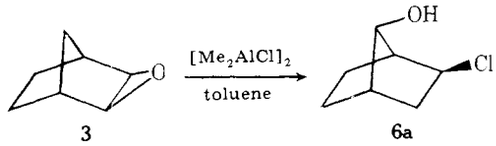
hand, allylic couplings decrease from the 2.0–3.5-Hz range found in most α-methylene γ-lactones[2,7,8] to values of 1 Hz or less in most α-methylene δ-lactones.[5,9]

In light of the above generalizations, a methylene doublet with a *geminal* splitting of 1.3 Hz, as in **4,** must be assigned



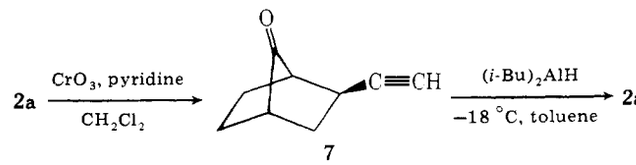
6a, X = Cl
b, X = Br



3 6a

to a methylene group in the α position on a δ-lactone. A plausible structure for **2** is thus **2a,**[10] obviously an excellent precursor for the δ-lactone **4.** The ethynylation of **3** by dimethylethynylaluminum etherate must then be proceeding by rearrangement under the influence of this Lewis acid reagent. Such substitution patterns (**6**) are well known as the products of the reaction of **3** with protic acids such as HBr and HCl.[11] The implication that they can also be formed with Lewis acid reagents[12] can be verified by noting that **3** gives **6a** when treated with $[(CH_3)_2AlCl]_2$.

Confirmation of structure **2a** is afforded by its oxidation to the ketone **7.** The IR spectrum of **7** shows split carbonyl bands



7

centered at high frequency (1775 cm^{-1}) characteristic of the strained bicyclo[2.2.1]heptan-7-one system.[13] Reduction of **7** by diisobutylaluminum hydride gives **2a** again, proving that the apical ketone arises from an apical hydroxyl in **2a** and not from rearrangement during oxidation.

The sequence **3** → **2a** → **4** thus represents a facile two-step synthesis of an unusual fused-ring α-methylene δ-lactone from commercially available starting materials.

Experimental Section

Infrared spectra were recorded on a Perkin-Elmer 283 and NMR spectra were recorded on Varian A-60 and XL-100 instruments using tetramethylsilane as internal standard. Mass spectra were obtained on an AEI MS-9. Gas chromatographic analyses were carried out on a Perkin-Elmer 3920.

Preparation of *exo*-2-Ethynylbicyclo[2.2.1]heptan-*syn*-7-ol (2a). To a solution of dimethylethynylaluminum etherate[5] in toluene (1 M, 45 mL) was added under nitrogen, while stirring, a solution of *exo*-2,3-epoxybicyclo[2.2.1]heptane (**3**) (2.2 g, 20 mmol, in 20 mL of toluene) at room temperature. After 2 h, the reaction mixture was hydrolyzed by the slow addition of a minimum amount of water. The solution was then dried ($MgSO_4$), filtered, and evaporated in vacuo. The residue was purified by TLC on silica gel plates (development with 4:1 C_6H_6/EtOAc) to give **2a** (0.9 g, 35%) as a low-melting solid, homogeneous by VPC (5% carbowax 20 M, 180 °C): IR (neat) 3400 (s), 3300 (s), and 2110 (m) cm^{-1}; NMR ($CDCl_3$) δ 3.98 (m, 1 H), 2.3–2.58 (m, 1 H), 2.18 (d, $J \approx 2$ Hz, 1 H), 0.9–2.1 (m, 8 H).

Anal. Calcd for $C_9H_{12}O$: *m/e* 136.08881. Found: 136.08876.

Preparation of Hexahydro-3-methylene-4,7-methanocyclopenta[*b*]pyran-2(3*H*)-one (4). To a mixture of palladium chloride (0.18 g, 1 mmol) and thiourea (0.07 g, 1 mmol), in acetone (5 mL) under 50 psi of carbon monoxide at 50 °C, was added a solution of **2a** (0.14 g, 1 mmol, in 3 mL of acetone), and the mixture was stirred for 48 h at that temperature. It was then filtered through a bed of celite and evaporated in vacuo. The residue was digested with water and was extracted with ether (3 × 20 mL). The combined organic layers were dried ($MgSO_4$) and evaporated in vacuo. The residue was then distilled at 0.1 mm pressure, and the fraction boiling at 62–65 °C was purified by TLC (4:1 C_6H_6/EtOAc) on silica gel plates to yield **4** as an oil (0.07 g, 47%); it was homogeneous by VPC (5% DEGS, 160 °C): IR (neat) 1722 (s), 1647 (m), and 1633 (w) cm^{-1}; NMR ($CDCl_3$) δ 5.94 (d, J = 1.3 Hz, 1 H), 5.28 (d, J = 1.3 Hz, 1 H), 4.50 (m, 1 H).

Anal. Calcd for $C_{10}H_{12}O_2$: C, 73.17; H, 7.32. Found: C, 72.67; H, 7.39.

Formation of *exo*-2-Chlorobicyclo[2.2.1]heptan-*syn*-7-ol (6a) from 3. To a stirred solution of dimethylaluminum chloride (5.5 mL, 11 mmol) (Texas Alkyls) in toluene, under nitrogen at room temperature, was added a solution of *exo*-2,3-epoxybicyclo[2.2.1]heptane (**3**) (1.1 g, 10 mmol) in toluene (6 mL) and stirred for 1 h. The mixture was then hydrolyzed with a minimum amount of water, dried ($MgSO_4$), filtered, and evaporated in vacuo. The product was purified by TLC (4:1 C_6H_6/EtOAc) on silica gel plates to give **6a** (1.2 g, 80%). Recrystallization from hexane gave crystals with mp 52–54 °C (lit.[11b]

52–53.2 °C). The IR and NMR spectra were identical with those reported for **6a**.[11b]

Preparation of *exo*-2-Ethynylbicyclo[2.2.1]heptan-7-one (7). Chromium trioxide (1 g, 10 mmol) was added to a stirred solution of pyridine (1.6 g, 20 mmol) in 25 mL of methylene chloride.[14] After 15 min at room temperature, 0.15 g (1.5 mmol) of **2** in 2 mL of methylene chloride was added, and the suspension was stirred at room temperature for 18 h, after which it was poured into water (20 mL) and filtered through a bed of celite. The organic layer was separated, and the aqueous layer was extracted with ether (3 × 20 mL). The combined organic layers were then washed with cold dilute hydrochloric acid and water, dried ($MgSO_4$), filtered, and evaporated in vacuo to give a yellow oil. This was distilled to give **7** (0.08 g, 50%), bp 50 °C (0.08 mm). It was homogeneous by VPC (5% carbowax 20 M, 160 °C): IR (neat) 3300 (s), 1830 (m), 1775 (s), and 1742 (m) cm^{-1}; NMR ($CDCl_3$) δ 2.86–2.43 (m, 1 H), 2.2 (d, J = 2 Hz, 1 H), 1.36–2.18 (m, 8 H). The 2,4-dinitrophenylhydrazone of **7** melted at 110–111 °C.

Anal. Calcd for $C_{15}H_{14}N_4O_4$: C, 57.32; H, 4.49; N, 17.83. Found: C, 57.27; H, 4.47; N, 17.73.

Reduction of 7 with Diisobutylaluminum Hydride to 2a. To a stirred solution of **7** (0.095 g, 0.8 mmol) in 1 mL of toluene at −18 °C was added a toluene solution of diisobutylaluminum hydride (1 mL, 2 M). Stirring was continued for 1 h at −18 °C. The reaction mixture was then hydrolyzed with a minimum amount of water, dried ($MgSO_4$), filtered, and evaporated in vacuo to give **2a** (0.09 g, 82%), which showed IR and NMR spectra identical with that of **2a** prepared earlier.

Acknowledgments. We thank Professor B. Snider for helpful discussions, Hoffmann-La Roche, Inc., for microanalytical services, and Matthey-Bishop, Inc., for a generous loan of $PdCl_2$. This investigation was supported by Grant CA 18546 and by training grants (to T.M. and V.V.) awarded by the National Cancer Institute, DHEW.

Registry No.—**2a,** 64130-75-2; **3,** 3146-39-2; **4,** 64130-76-3; **6a,** 16709-78-7; **7,** 64130-77-4; 7 DNP, 64130-78-5.

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Synthesis and Circular Dichroism Spectral Studies of Arylamides of *trans*-2-Phenylcyclohexanecarboxylic Acid and *trans*-1-Amino-2-phenylcyclohexane[1,2]

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November 18, 1976

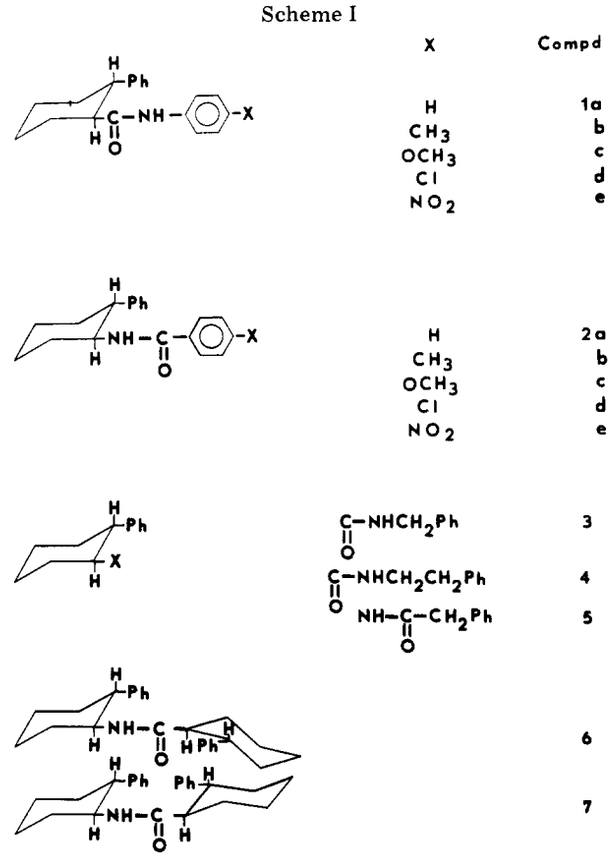
Although the chiroptical phenomena exhibited by aromatic compounds have been studied extensively,[3] papers on benzamides are few,[4–6] and a systematic study of anilides has not been reported. We report the CD and isotropic UV spectra of a series of aromatic amides (Scheme I) derived from *trans*-2-phenylcyclohexanecarboxylic acid (A) and *trans*-1-amino-2-phenylcyclohexane (B).

These amides are of interest because they have structural features in common with amides of the amino acid phenylalanine. The C-1 and C-2 substituents in these amides form a fixed dihedral angle of approximately 60°, resulting in a chromophoric system which resembles a staggered conformer of the analogous phenylalanine amides. Absolute configurational assignments and conformational analysis are available from previously reported studies on the precursors, A and B.[7,8] Finally, the CD spectra of these amides show *separately* the effects of changing the para substituent on an anilide or benzamide, inverting the amide chromophore, or changing the proximity (number of intervening carbons) of the amide and benzene chromophores.

Experimental Section

CD and ORD measurements were made at 25 °C in methanol on a Jasco Model ORD/UV/CD-5 instrument under conditions described by Verbit et al.[7,9] Isotropic UV measurements were made on a Cary Model 11 instrument. For CD, ORD, and isotropic UV measurements, solution concentrations were 2.5–3.0 × 10^{-4} M, except that the $[\alpha]_D$'s of all compounds and the ^1L_b bands (CD and UV) of compounds **3–7**

Scheme I



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